

# CHAPTER 3

## EXPERIMENTAL

### 3.1 Materials

Throughout this research, commercial materials used for the characterisation are Technyl® A216 (unreinforced PA 6,6), Celanese® 1503-2, short glass fibre reinforced PA 6,6 composite, (33% fibre by weight), Technyl® A216 V30 NAT, short glass fibre reinforced PA 6,6 composites, (30% fibre by weight) and LNP Thermocomp RC 100-10, short carbon fibre reinforced PA 6,6 composite, (44% fibre by weight).

### 3.2 Sample preparation

#### 3.2.1 Glass fibre composites

Glass fibre composite with 18%  $V_f$  which is 33% fibre by weight, were prepared from short glass fibre reinforced PA 6,6 composite, manufactured by Hoechst Celanese Corporation, USA. The materials were used as received. Glass fibre composite with 16%  $V_f$  which is 30% fibre by weight, were prepared from short glass fibre reinforced PA 6,6 composite, manufactured by Rhodia Polyamide Co., Ltd., Korea which used as received.

On the other hand, glass fibre composite with 13% , 8% and 4%  $V_f$  has been prepared by diluting Technyl® A216 V30, short glass fibre reinforced PA 6,6 composites, (30% fibre by weight) with unreinforced PA 6,6 (Technyl® A216 ) manufactured by Rhodia Polyamide Co., Ltd., Korea.

### 3.2.2 Carbon fibre composites

Carbon fibre composites with volume fraction of 33% were used as received. The materials used were carbon fibre composite (LNP Thermocomp RC 100-10) manufactured by SABIC Innovative Plastics.

### 3.2.3 Short glass/short carbon hybrid fibre composites

Preparation of short glass/short carbon hybrid fibre composites were done by using carbon fibre composite (LNP Themocomp RC100-10) and short glass fibre reinforced PA 6,6 composite, (33% fibre by weight). These two materials were mixed together in different ratios to form type A hybrid fibre (dispersed fibre) as mentioned previously in section 1.3 (chapter one).

Physical blending approach has been used to mix those materials. Before the moulding process, the composites were dried in a vacuum oven at 90°C for at least eight hours. Finally, all the blends were prepared in batches with total weight of 500 g per batch to prevent some compounded composite materials from settled down at the bottom part in the feed hopper of the injection moulding machine due to the difference in material density.

### 3.3 Preparation of moulded specimens

In this approach, composites with eight different fibre volume fractions were prepared. All the substances were shaken together in a plastic bag for about 10 minutes to mix them before feeding them into the hopper. A single gated double cavity, impact and tensile standard test bar mould was used in the moulding. The dimensions of dumb-bell shaped tensile test pieces were in accordance with the ASTM Standard D638-80,

type 1 [51]. The list of materials was given in Table 3.1. Temperature settings on automatic injection moulding machine model Boy® 50M were listed in Table 3.2.

### 3.4 Determination of fibre volume fraction in composites

#### 3.4.1 Fibre extraction

Heat digestion method was used to determine fibre volume levels of glass fibre composites since it has good resistance to heat. Nevertheless, the temperature required for glass fibre to burn off could cause oxidation of carbon fibre since carbon fibre starts to oxidised above 400°C [52]. This behaviour will give an inaccurate value in the determination of fibre volume fraction. Therefore, acid digestion method was used.

##### 3.4.1.1 Carbon fibre composites

For the carbon fibre composite samples, fibre volume fraction,  $V_f$  was determined by acid digestion method. Samples were cut from the central portion of injection moulded test pieces, accurately weighed and placed in a 500 ml beaker. Concentrated sulphuric acid was added to dissolve the polyamide matrix. Then, the residual fibres were collected by filtration through a sintered glass crucible. The extracted fibres were then washed with distilled water, dried in a vacuum oven at 100°C for four hours, cooled and dried in desiccators and then weighed.

##### 3.4.1.2 Glass fibre composites

Volume fraction,  $V_f$  for glass fibre composites sample was determined by heat treatment. Samples from the central portion of injection moulded test pieces were cut and the polymer matrix was removed by heating the specimen in a muffle furnace at a temperature of up to 700°C for six to seven hours. Fibres were then weighed.

### 3.4.1.3 Short glass/short carbon hybrid fibre composites

Fibre volume fraction in hybrid fibre composites was determined by calculation since strong acids can corrode glass fibre and carbon fibre starts to oxidise at a temperature above 400°C [52]; neither acid nor heat digestion method can be used in the extraction of fibres.

## 3.4.2 Calculations

### 3.4.2.1 Single glass and carbon fibre composites

Fibre volume fraction,  $V_f$  was calculated by equation (3.1) for single glass and carbon fibre composites:

$$V_f = \frac{\frac{M_f}{\delta_f}}{\frac{M_f}{\delta_f} + \frac{M_m}{\delta_m}} \quad (3.1)$$

where  $M$  and  $\delta$  are weight and density, respectively; and subscripts  $f$  and  $m$  refer to the fibre and matrix, respectively.

### 3.4.2.2 Short glass/short carbon hybrid fibre composites

Fibre volume fraction  $V_f$  was calculated by equation (3.6) for short glass/short carbon hybrid fibre composites:

$$V_m = \frac{M_m}{\delta_m} \quad (3.2)$$

$$V_{gf} = \frac{M_{gf}}{\delta_{gf}} \quad (3.3)$$

$$V_{cf} = \frac{M_{cf}}{\delta_{cf}} \quad (3.4)$$

$$V_{(t)} = V_{(m)} + V_{(cf)} + V_{(gf)} \quad (3.5)$$

$$V_f = \frac{V_{(gf)} + V_{(cf)}}{V_{(t)}} \quad (3.6)$$

where V is refer to volume; subscripts gf and cf refer to the glass fibre and carbon fibre, respectively.

### 3.5 Specimen conditioning

#### 3.5.1 Dry

Specimens in dry condition refer to specimens as it was moulded. The specimens were dried in vacuum oven for four hours at 90°C to make sure no water exists in the specimens. Then the specimens were cooled and put into vacuumed desiccators with silica gel as an indicator to avoid any moisture from the environment.

#### 3.5.2 50% Relative Humidity

The test pieces were conditioned to 50% relative humidity (50% RH) at 23°C. The specimens were placed in a desiccator with the atmosphere of saturated sodium hydrogen sulphate (NaHSO<sub>4</sub>.H<sub>2</sub>O) solution for one month prior to the testing [53].

#### 3.5.3 Wet

Hot water bath was used to prepare wet condition of the specimens. The specimens were boiled for 24 hours to ensure the specimens completely wet. After 24 hours of boiling, the specimens were kept in wet environment by keeping the specimens in a basin filled with distilled water at room temperature.

### 3.6 Determination of fibre length distribution

Samples were cut about 1 cm length from the central portion of injection moulded test pieces. Fibres used in this procedure were recovered from composite specimens using the methods as described in section 3.4.1.1 and 3.4.1.2 for carbon fibre composites and glass fibre composites respectively.

Then the fibres were immersed in a large beaker containing a microscope glass slide, distilled water and a few drops of detergent to reduce surface tension. The solution was placed in an ultrasonic bath for about 45 to 90 minutes to mix the fibres uniformly. The solution was then removed from the beaker using a dropper, leaving a small amount of solution with fibres on the glass slide. The slides with fibres on one side were dried in an oven for two hours.

Image Analyser model Leco, IA-32, connected to Dell computer was used to measure the fibre length. At least five hundred fibres were counted for each batch of specimen. Data processing involved rearranging fibre length in ascending order using excel programme and dividing the fibre percentage according to fibre length range. Eventually, the histogram of percentage of fibre against fibre length range was plotted.

### 3.7 Determination of thermal properties

#### 3.7.1 Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was used to investigate thermal decomposition behaviour of materials with a TGA 6 Thermogravimetric Analyzer (Perkin Elmer, U.S.A.). Tests were done under nitrogen at a scan rate of 20°C/min in a programmed temperature range of 50°C to 900°C. A sample of 5 mg to 10 mg was used for each run. The weight change was recorded as a function of temperature. Derivative

peak temperature (DTp) was taken as the maximum temperature acquired from the differentiation of the weight change as a function of time.

### 3.7.2 Differential scanning calorimetry (DSC)

Differential scanning calorimetry (DSC) experiments were performed with a Diamond DSC (Perkin Elmer, U.S.A.). Each sample was heated and cooled at a scanning rate of 5°C/min under nitrogen atmosphere in order to prevent oxidation. A test sample of 5 mg to 10 mg was placed in an aluminium pan and heated from 0°C to 200°C then cooled from 200°C to 0°C for each run. The melting temperature ( $T_m$ ), enthalpy ( $\Delta H_m$ ), crystallisation temperature ( $T_c$ ), crystalline enthalpy ( $-\Delta H_m$ ) were determined after the melt-crystallisation process.

### 3.7.3 Dynamic mechanical analysis (DMA)

The dynamical mechanical analysis was carried out to investigate the effects of temperature and frequency on viscoelastic properties of polymer and polymer composites [54]. The dynamic mechanical properties of glass fibre reinforced PA 6,6 were analysed with a DMA Q800 (TA Instrument Inc. New Castle, Delaware, U.S.A.). DMA test specimens were taken from the middle section of the moulded dumb-bell bar. DMA specimens cut from moulding samples were rectangular strips with dimensions of 60 mm x 13 mm x 3.3 mm and subjected to three-point bending with a span length of 35 mm. Measurements were conducted over the temperature range of -100°C to 200°C with a heating rate of 3°C/min, and under a constant frequency of 1.0 Hz. By measuring the time lag of the displacement to the applied force, the damping factor of the material was determined.

## 3.8 Determination of mechanical properties

### 3.8.1 Tensile testing

The tensile tests were performed using an INSTRON Universal Testing (model 5569) with a constant crosshead speed of 1.0 mm/min at room temperature of about 23°C with a sample rate of 10.0 pts. The injection moulded dumb-bell shaped specimens were used to perform the test. The distance between jaws of the clamps (grip distance) was adjusted and fixed at 95 mm. The test specimen was then clamped in the testing machine and the test was conducted. All the data and calculations were transferred to the computer and the graphs were plotted. ASTM standard D638-80 [51] was used as a standard in calculating the tensile properties.

For every batch of specimens, the result reported for at least four tests on average. The dimensions of dumb-bell shaped injection moulded tensile test specimen were given in Figure 3.1.

### 3.8.2 Flexural testing

In this work, three point bending flexural test was carried out using an INSTRON Universal Testing (model 5569) at room temperature of about 23°C at a constant cross-head speed of 1.28 mm/min with sample rate of 10.0 pts. The speed or rate of crosshead motion,  $R$  was calculated using the equation

$$R = \frac{ZL^2}{6d} \quad (3.7)$$

where  $L$  and  $d$  are the specimen support span and depth of specimen respectively. The thickness of the sample became depth because the specimens were mounted in the flat position.  $Z$  is the rate of straining of the outer fibre (equal to 0.01). Specimen support span,  $L$  was fixed at 50 mm.



ASTM D790 [55] was used as a standard to calculate flexural strength,  $\sigma_f$  and flexural modulus,  $E_b$ . For every batch of specimens, the results reported are the average of at least four tests. Figure 3.2 shows the specimen arrangement during the test.

### 3.8.3 Impact testing

Impact test in Charpy mode was conducted in this study. It measures the energy absorbed by a standard notched specimen while breaking under an impact load. The Charpy impact test continues to be used as an economical quality control method to determine the notch sensitivity and impact toughness of engineering materials.

Impact test bars were with dimension of 6 mm x 12 mm x 127 mm and cut into two pieces with length of about 64 mm. The test bars were notched at the centre on one edge to produce a single edge notch (SEN) specimen. Specimens were notched with four different notches to depth ratios ( $a/D$ ) i.e. 0.1, 0.2, 0.3 and 0.4 for each batch by using the *Ray-Ran* notch cutter machine. Throughout the test, a support span to depth ratio ( $S/D$ ) was maintained at 4 [34]. Figure 3.3 show the single edge notch (SEN) impact test specimen.

The impact test was conducted using an INSTRON Dynatup model 9210 impact tester. The impact is applied on the centre of the notch by a cylindrical steel projectile of 1.2 kg mass. This machine is equipped with a 6.448 kg load cell and the test height of 0.226 m was set. For the Charpy test, specimens were supported on the Charpy fixture with a span of 48 mm. The striking tup struck the specimen midway between the supports. At least five specimens were tested for every batch of material.

### 3.9 Microscopic study

Fractured surfaces were examined using Field Emission Auger Microprobe model JAMP-9500F. SEM micrographs were taken at 10 keV acceleration voltage at various magnifications. The fracture parts of specimens were sputter-coated with a thin layer of gold in order to improve the sample conductivity and to avoid electrical charging during examination.